

REPORT DOCUMENTATION PAGES

JUN 2 5 1981

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, If different from Report)

To be published in the Journal of Prosthodontic Dentistry

18. SUPPLEMENTARY NOTES

DD 1 JAN 73 1473

Unlimited

1. REPORT NUMBER

UNCLASSIFIED

READ INSTRUCTIONS

PERIOD COVERED

BEFORE

2. SOVE ACCESSION NO. 3. RECIPIENT'S CATALUS HUMBER

13. KEY YORDS (Continue on reverse side it necessary and identity by block number)

Resins, denture resins, polymethylmethacrylates, fiber reinforcement and fatigue failure.

29. ABSTRACT (Continue on reverse side it necessary and identity by block number) The effect of carbon fiber reinforcement on the mechanical properties and flexural fatigue resistance of three proprietary denture resins was evaluated. Incorporation of chopped carbon fibers resulted in increases in fatigue resistance of 16 to 83 percent. Pretreatment of the fibers with a silane coupling agent resulted in values for fatigue resistance that were 42 to 100 percent higher than those observed for the unreinforced resins. Carbon fiber reinforcement did not significantly alter the mechanical properties of the test materials.

----

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entere

Polymethylmethacrylate reinforcement: Affect on fatigue failure.

Dennis R. Skirvin, B.S., D.D.S.

Stanley G. Vermilyea, B. S., D.M.D., M.S.

Robert E. Brady, B.S., D.D.S. \*\*\*

U.S. Army Dental Activity and U.S. Army Institute of Dental Research,

Walter Reed Army Medical Center, Washington, D.C., 20012.

Commerical materials and equipment are identified in this report to specify the experimental procedure. Such identification does not imply official recommendation or endorsement or that the materials and equipment are necessarily the best available for the purpose. Furthermore, the opinions expressed herein are those of the authors and are not to be construed as those of the Army Medical Department.

Availability Code

Avail and/er
Dist Special

<sup>\*</sup> Major, Senior Resident, Removable Prosthodontic Service 11 24.

<sup>\*\*</sup> Lieutenant Colonel, Chief, Division of Dental Materials ...tirication....

<sup>\*\*\*</sup> Colonel, Chief, Removable Prosthodontic Service.

Acrylic resins have been the materials of choice for the fabrication of denture bases for over forty years. 1 Esthetics, simple processing techniques and relative ease of repair have been largely responsible for the routine use of these materials. However, the acrylic resins are not totally devoid of undesirable characteristics. Susceptibility to fracture from impact, masticatory tensile and shear forces, or cyclic fatigue appears to be a limiting factor in the routine use of these materials. 1,2 Several studies have demonstrated that the flexure of maxillary and mandibular complete denture prostheses may exceed 1.5 mm at the midline. 3,4 At least one study 5 estimated that complete dentures undergo flexure about 500,000 times over the course of a year and another 6 has suggested that resistance to flexural fatigue may be the most significant mechanical property for predicting the useful clinical life of denture base polymers.

Alternatives to poly(methylmethacrylate) resins such as epoxy, polycarbonate, and polyvinyl materials as well as reinforcement of the polymethyl methacrylate materials with wires, nylon mesh, fiberglass, silicon carbide, aluminum oxide, sapphire fibers and carbon fibers have been employed to enhance the longevity of the denture base. Results of investigations on the use of reinforcing agents have ranged from a decrease in strength to a significant strength improvement. Carbon fiber reinforcement of poly(methylmethacrylate) specimens has been shown to provide significant increase in the impact strength and transverse strength. However, data as to the fatigue resistance of carbon reinforced poly(methylmethacrylate) is not available.

The present study was undertaken to evaluate the tensile properties and resistance to flexural fatigue of three commercially available denture base resins when plain and silanized carbon fibers were employed as reinforcing agents.

#### MATERIALS AND METHODS

# Preparation of test specimens

The denture resins (Lucitone, \*Duraflow and Hi-I ) were obtained from proprietary sources. Carbon fibers were obtained as long (90 mm) strands and reduced manually into 3-4 mm segments. Some of the segments were coated with a commercially available silane coupling agent \*\* prior to incorporation into the resin.

Specimens for the determination of flexural fatigue were 10 mm X 65 mm X 2.5 mm strips prepared as prescribed by American Dental Association Specification No. 12 for the transverse deflection test of denture base resins. Specimens for the determination of tensile properties were 6 mm X 70 mm rods. The ends of the rods were flared to a diameter of 16 mm to facilitate stabilization and alignment during tensile loading. The resins

<sup>\*</sup> The L. D. Caulk Co., Division of Densply International, Milford, DE 19963.

<sup>+</sup> Product Research Laboratories, Cambridge, MA 02139

<sup>#</sup> Fricke Dental Manufacturing Co., Villa Park, IL 60181.

<sup>§</sup> Hercules Incorporated Salt Lake City, UT 84125.

<sup>\*\*</sup> A-174 Silane, Union Carbide Corporation, New York, NY 10017.

were mixed and packed in accordance with their respective manufacturers' instructions. Carbon fibers (0.40 percent by weight) were dispersed in the polymer prior to monomer addition for the preparation of reinforced test specimens. The packed molds were processed at 74° C for nine hours and permitted to cool to room temperature prior to divestment of the test pieces.

Five specimens were fabricated for each material under each condition for flexure fatigue determination. A like number of specimens were constructed for the evaluation of tensile properties. All specimens were stored in distilled water maintained at  $37^{\pm}2^{\circ}$  C for two weeks prior to testing. All tests were conducted at  $23^{\pm}2^{\circ}$  C and  $50^{\pm}10$  percent relative humidity. Determination of properties

The specimens for the determination of fatigue resistance were placed in a three-point loading device affixed to a dynamic testing machine<sup>++</sup> (Fig. 1). An initial loading of 0.4 Kg was applied to the center of each specimen. Then the load was increased to 5.4 Kg and returned to the initial value at a rate of 330 cycles per minute. The number of cycles completed to fracture was recorded as an indicator of resistance to flexural fatigue.

Tensile properties were determined on a constant displacement rate testing machine \*\* at a crosshead speed of 0.02 inch per minute. Elongation was measured over a one-inch gage length with a breakaway extensometer. Self aligning fixtures (Fig. 2) were employed to ensure proper placement of the specimens with respect to the direction of the applied force.

<sup>++</sup> Instron Model 1350, Instron Corporation, Caonton, MA 02021.

<sup>§§</sup> Extensometer Model G 51-12, Instron Corporation, Canton, MA 02021.

#### **RESULTS**

Data on the fatigue resistance of plain and reinforced denture base polymers are summarized in Fig. 3. The highest and lowest values for the number of cycles to failure for each resin under each condition were discarded to reduce the variability in the data and the mean values and standard deviations calculated. The range of the resistance to flexural fatigue was deline at ed by observations on unreinforced Duraflow (1,300 cycles) and Hi-I reinforced with silanized carbon fibers (12,000 cycles). Addition of carbon fibers to the denture base resins increased the number of cycles required to elicit specimen failure by 16 percent, 33 percent and 83 percent for Duraflow, Lucitone and Hi-I respectively over that observed with the resins in the unreinforced condition. Reinforcement of Duraflow, Lucitone and Hi-I with silanized carbon fibers yielded respective increases in fatigue resistance of 42 percent, 48 percent and 100 percent over the unreinforced materials.

Data on the apparent mechanical properties of the test resins are summarized in the Table. The employment of the carbon fibers or silanized carbon fibers neither increased nor decreased the apparent properties of the denture base resins.

## DISCUSSION

From the available data, it would appear that carbon fibers randomly dispersed in a denture base polymer prior to processing significantly increase the fatigue resistance of the resin without affecting its apparent mechanical properties. Additional resistance of the resins to fatigue failure is realized if the fibers are treated with a silane coupling agent prior to incorporation into the denture base polymer.

It would appear that the use of the silane increases the surface activity of the carbon fibers and facilitates stress transfer from the poly(methylmethacrylate) to the carbon fiber.

The lack of augmentation or deterioration of the apparent mechanical properties with carbon reinforcement as well as the wide variation in the cyclic fatigue data may be due to random dispersion of the reinforcing fibers in the polymerized resins or the polymer-fiber ratio. Other studies 10,12 have indicated a reduction of transverse strength values of denture base resins when untreated carbon fibers were used as the reinforcing agent. In this study, no attempt was made to vary the polymer-fiber ratio or control the orientation of the fibers within the processed polymers. Control of these factors may indicate the efficacy of the use of silanized carbon fibers for further enhancement of the strength and fatigue resistance of poly(methylmethacrylate) denture base resins. Further studies designed to address the effects of fiber concentration and orientation are indicated.

The obvious cosmetic effects of carbon fibers on the completed denture suggest their confinement to the palatal and lingual aspects of maxillary and mandibular prostheses. A technique has been described for accomplishment of this goal and the use of carbon reinforcement may provide a low cost alternative to cast metal based dentures to reduce the incidence of midline fatigue fracture. Furthermore, the increased resistance to fatigue failure afforded by carbon fiber reinforcement of denture base resins would be invaluable for cases requiring a soft relining material with a concomitant reduction in the thickness of the hard denture base.

#### SUMMARY

The effect of carbon fiber reinforcement on the mechanical properties and flexural fatigue resistance of three proprietary denture resins was evaluated. Incorporation of chopped carbon fibers resulted in increases in fatigue resistance of 16 to 83 percent. Pretreatment of the fibers with a silane coupling agent resulted in values for fatigue resistance that were 42 to 100 percent higher than those observed for the unreinforced resins. Carbon fiber reinforcement did not significantly alter the mechanical properties of the test materials.

## LEGENDS FOR FIGURES

- Figure 1. Device for application of cyclic three point loading.
- Figure 2. Self aligning fixture employed during the application of tensile forces.
- Figure 3. Results of cyclic fatigue tests.
  - A) Duraflow; B) Duraflow + carbon fibers; C) Duraflow + silanized carbon fibers; D) Lucitone; E) Lucitone + carbon fibers; F) Lucitone + silanized carbon fibers;
     G) Hi-I; H) Hi-I + carbon fibers and I) Hi-I + silanized carbon fibers.

### REFERENCES

- 1. Hargreaves, A. S. The prevalence of fractured dentures. Br Dent J 126: 451-455 1969.
- 2. Waters, N. E. The fatigue fracture of acrylic. <u>Dent Pract Dent Rec 18:</u> 389-392 1968.
- Regle, C. P., and Kydd, W. L. Preliminary study of the lateral deformation of metal base dentures in relation to plastic base dentures.
   J Prosthet Dent 3:326, May 1953.
- 4. Lambrecht, J. R., and Kydd, W. L. Functional stress analysis of the maxillary complete denture base. J Prosthet Dent 12:865 Sep-Oct 1962.
- 5. Johnson, Wesley, and Mathews, Ernest. Fatigue studies on some dental resins. Brit Dent J 86:252 May 1949.
- 6. Smith, D. C. Acrylic denture base; mechanical evaluation of dental polymethylmethacrylate. Brit Dent J 111:9 July 1961.
- 7. Kydd, W. L., and Wykhuis, W. A. A modified epoxy resin as a denture base material. <u>JADA 56</u>:385 Mar 1958.
- 8. Jennings, R. E., and Wuebbenhorst, A. M. The effect of metal reinforcements on the transverse strength of acrylic resin. <u>J Dent Child 27</u>: 162, 1968.
- 9. Grant, A. A., and Greener, E. H. Whisker reinforcement of polymethyl methacrylate denture base resins. Austral Dent J 12:29 Feb 1967.
- Schreiber, C. K. Polymethylmethacrylate reinforced with carbon fibers.
   Brit Dent J 130:29 Jan 1971.
- 11. Bowen, R. L. Effect of particle shape and size distribution in a reinforced polymer, JADA 69:481 Oct 1964.

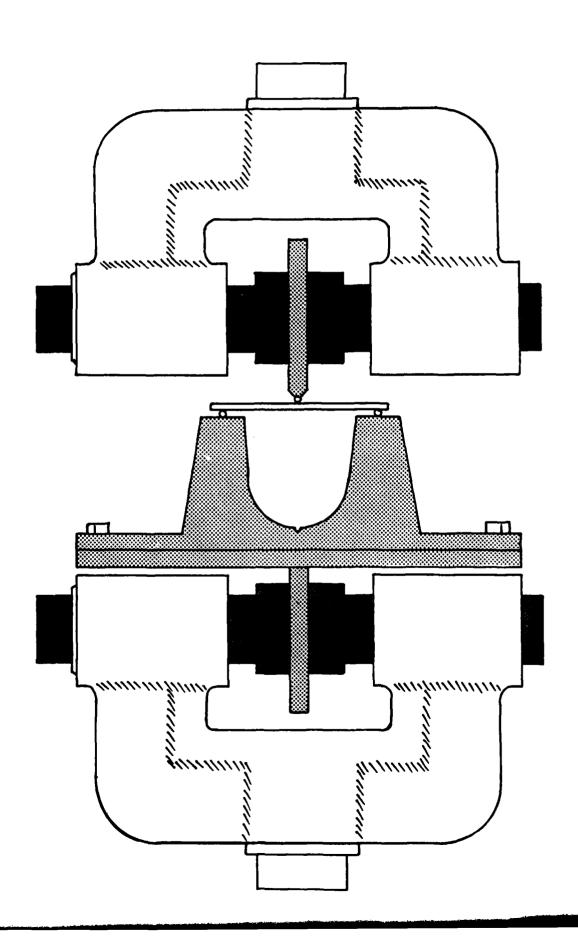
- 12. Wylegala, R. T. Reinforcing denture base material with carbon fibers.

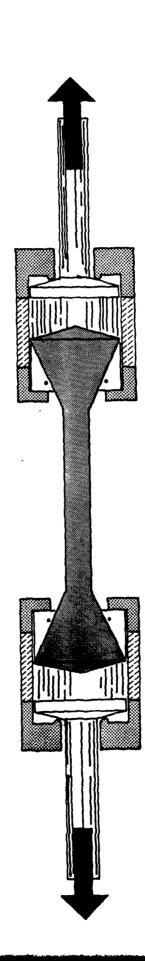
  Dent Tech 26:97-100, 1973.
- 13. <u>Guide to Dental Materials and Devices</u>, ed 7, American Dental Association, Chicago, 1974-1975, pp 203-209.

Table. The effect of carbon fiber reinforcements on the apparent mechanical properties of denture base resins

0000	* CU Est C				
Material	Ultimate * Tensile Strength (psi)	Yield Strength (0.2% Offset) (psi)	Elastic Limit * (psi)	Modulus of* Elasticity (X10 <sup>5</sup> psi)	Elongation (%)
Duraflow					
Plain	8,200 (250)	5,400 (400)	3,400 (500)	4.06 (0.21)	3.3 (0.4)
Carbon fibers	7,600 (300)	6,800 (400)	4,300 (800)	4.57 (0.38)	2.0 (0.2)
Silanized carbon fibers	7,600 (500)	6,000 (400)	3,400 (300)	4.63 (0.33)	1.8 (0.5)
Lucitone				:	
Plain	7,800 (900)	5,200 (900)	2,600 (800)	4.4 (1.3)	3.4 (0.3)
Carbon fibers	7,800 (900)	6,100 (700)	3,700 (700)	4.4 (0.4)	2.4 (0.8)
Silanized carbon fibers	8,100 (500)	6,400 (300)	3,600 (200)	4.6 (0.2)	2.6 (0.5)
Hi-I					
Plain	7,500 (200)	4,900 (200)	2,900 (400)	4.1 (0.2)	3.3 (0.2)
Carbon fibers	7,300 (200)	5,700 (400)	4,000 (500)	4.0 (0.4)	3.0 (0.6)
Silanized carbon fibers	7,900 (500)	6,300 (600)	4,200 (500)	3.9 (0.2)	2.5 (0.7)

<sup>\*</sup> Means with standard deviations in parenthesis.





NUMBER OF CYCLES TO FAILURE (X 10-3)

